

### CERTIFICATION

SDG No: MC45838A Laboratory: Accutest, Massachusetts  
Site: BMS, Building 5 Area, PR Matrix: Soil  
Humacao, PR

**SUMMARY:** Soil samples (Table 1) were collected on the BMSMC facility – Building 5 Area. The BMSMC facility is located in Humacao, PR. Samples were taken May 10, 2016 and were analyzed in Accutest Laboratory of Marlborough, Massachusetts that reported the data under SDG No.: MC45838. Results were validated using the following quality control criteria of the methods employed (MADEP VPH and MAPED EPH, Massachusetts Department of Environmental Protection, 2004) and the latest validation guidelines (July, 2015) of the EPA Hazardous Waste Support Section. The analyses performed are shown in Table 1. Individual data review worksheets are enclosed for each target analyte group. The data sample organic data samples summary form shows for analytes results that were qualified.

In summary the results are valid and can be used for decision taking purposes.

Table 1. Samples analyzed and analysis performed

| SAMPLE ID | SAMPLE DESCRIPTION | MATRIX | ANALYSIS PERFORMED                                |
|-----------|--------------------|--------|---|
| MC45838-2 | RA-20 (6.5-7.5)    | Soil   | Volatiles TPHC Ranges;<br>Extractable TPHC Ranges |

Reviewer Name: Rafael Infante  
Chemist License 1888

Signature:



Date:

June 13, 2016



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## Report of Analysis

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|                          |                                     |                        |          |
|--------------------------|-------------------------------------|------------------------|----------|
| <b>Client Sample ID:</b> | RA-20 (6.5-7.5)                     | <b>Date Sampled:</b>   | 05/10/16 |
| <b>Lab Sample ID:</b>    | MC45838-2                           | <b>Date Received:</b>  | 05/11/16 |
| <b>Matrix:</b>           | SO - Soil                           | <b>Percent Solids:</b> | 74.8     |
| <b>Method:</b>           | MADEP VPH REV 1.1                   |                        |          |
| <b>Project:</b>          | BMSMC, Building 5 Area, Puerto Rico |                        |          |

| Run #  | File ID   | DF | Analyzed | By | Prep Date | Prep Batch | Analytical Batch |
|--------|-----------|----|----------|----|-----------|------------|------------------|
| Run #1 | AB94041.D | 1  | 05/12/16 | AF | n/a       | n/a        | GAB5168          |
| Run #2 |           |    |          |    |           |            |                  |

| Run #  | Initial Weight | Final Volume | Methanol Aliquot |
|--------|----------------|--------------|------------------|
| Run #1 | 16.6 g         | 16.0 ml      | 100 ul           |
| Run #2 |                |              |                  |

## Volatile TPHC Ranges

| CAS No. | Compound                    | Result | RL   | MDL  | Units | Q |
|---------|-----------------------------|--------|------|------|-------|---|
|         | C5- C8 Aliphatics (Unadj.)  | ND     | 8100 | 4100 | ug/kg |   |
|         | C9- C12 Aliphatics (Unadj.) | ND     | 8100 | 4100 | ug/kg |   |
|         | C9- C10 Aromatics (Unadj.)  | ND     | 8100 | 4100 | ug/kg |   |
|         | C5- C8 Aliphatics           | ND     | 8100 | 4100 | ug/kg |   |
|         | C9- C12 Aliphatics          | ND     | 8100 | 4100 | ug/kg |   |

| CAS No. | Surrogate Recoveries   | Run# 1 | Run# 2 | Limits  |
|---------|------------------------|--------|--------|---------|
|         | 2,3,4-Trifluorotoluene | 89%    |        | 70-130% |
|         | 2,3,4-Trifluorotoluene | 91%    |        | 70-130% |



ND = Not detected      MDL = Method Detection Limit  
 RL = Reporting Limit  
 E = Indicates value exceeds calibration range

J = Indicates an estimated value  
 B = Indicates analyte found in associated method blank  
 N = Indicates presumptive evidence of a compound

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## Report of Analysis

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|                          |                                     |                        |          |
|--------------------------|-------------------------------------|------------------------|----------|
| <b>Client Sample ID:</b> | RA-20 (6.5-7.5)                     | <b>Date Sampled:</b>   | 05/10/16 |
| <b>Lab Sample ID:</b>    | MC45838-2                           | <b>Date Received:</b>  | 05/11/16 |
| <b>Matrix:</b>           | SO - Soil                           | <b>Percent Solids:</b> | 74.8     |
| <b>Method:</b>           | MADEP EPH REV 1.1 SW846 3546        |                        |          |
| <b>Project:</b>          | BMSMC, Building 5 Area, Puerto Rico |                        |          |

| Run #  | File ID   | DF | Analyzed | By | Prep Date | Prep Batch | Analytical Batch |
|--------|-----------|----|----------|----|-----------|------------|------------------|
| Run #1 | DE14343.D | 1  | 05/31/16 | AP | 05/18/16  | OP47552    | GDE801           |
| Run #2 |           |    |          |    |           |            |                  |

| Run #  | Initial Weight | Final Volume |
|--------|----------------|--------------|
| Run #1 | 11.3 g         | 2.0 ml       |
| Run #2 |                |              |

## Extractable TPHC Ranges

| CAS No. | Compound                   | Result | RL    | MDL   | Units | Q |
|---------|----------------------------|--------|-------|-------|-------|---|
|         | C11-C22 Aromatics (Unadj.) | ND     | 24000 | 19000 | ug/kg |   |
|         | C9-C18 Aliphatics          | ND     | 12000 | 9500  | ug/kg |   |
|         | C19-C36 Aliphatics         | ND     | 12000 | 9500  | ug/kg |   |
|         | C11-C22 Aromatics          | ND     | 24000 | 19000 | ug/kg |   |

| CAS No.   | Surrogate Recoveries | Run# 1 | Run# 2 | Limits  |
|-----------|----------------------|--------|--------|---------|
| 84-15-1   | o-Terphenyl          | 75%    |        | 40-140% |
| 321-60-8  | 2-Fluorobiphenyl     | 72%    |        | 40-140% |
| 580-13-2  | 2-Bromonaphthalene   | 67%    |        | 40-140% |
| 3386-33-2 | 1-Chlorooctadecane   | 99%    |        | 40-140% |



ND = Not detected      MDL = Method Detection Limit  
 RL = Reporting Limit  
 E = Indicates value exceeds calibration range

J = Indicates an estimated value  
 B = Indicates analyte found in associated method blank  
 N = Indicates presumptive evidence of a compound

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## CHAIN OF CUSTODY

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 500 Korte Way, Dayton, OH 45424  
 TEL: 732-325-0200 FAX: 732-325-0201  
 www.accutest.com

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State Order Control # MC45838A  
 Get From NJ 0126

| Client / Reporting Information                   |  | Project Information   |  | Billing Information (if different from Report to)           |  | Matrix Codes |  |
|--|--|---|--|---|--|--------------|--|
| Company Name<br>Anderson Mulholland Associates   |  | Project Name<br>BMS Release Assessment  |  | Billing Information (if different from Report to)           |  | Matrix Codes |  |
| Street Address<br>2700 Westchester               |  | City<br>Purchase NY   |  | Company Name  |  | Matrix Codes |  |
| Project Contact<br>Terry Taylor                  |  | Project #   |  | Street Address  |  | Matrix Codes |  |
| Phone<br>914-251-0100                            |  | Client Purchase Order #   |  | City  |  | Matrix Codes |  |
| Email<br>N.Rivera, T.Taylor, D.Holmes            |  | Project Manager   |  | State   |  | Matrix Codes |  |
| Field ID / Point of Collection                   |  | Date  |  | Time  |  | Matrix Codes |  |
| 1 S-35D  |  | 5/11/16   |  | 1524  |  | NR 6W 5 5    |  |
| 2 RA-20 (6.5-7.5)                                |  | 5/10/16   |  | 1450  |  | TY 50 5 5    |  |
| 3 S-41D  |  | 5/10/16   |  | 1418  |  | NR 6W 5 5    |  |
| 4 S-42D  |  | 5/10/16   |  | 1420  |  | NR 6W 5 5    |  |
| 5 RA6-GWS  |  | 5/19/16   |  | 1645  |  | TY 6W 3 3    |  |
| 6 RA6-GWS  |  | 5/19/16   |  | 0950  |  | TY 6W 1 1    |  |
| 7 RA20-GWS                                       |  | 5/10/16   |  | 1400  |  | TY 6W 5 5    |  |
| Turnaround Time (Business days)                  |  | Date Deliverable Information  |  | Comments / Special Instructions                             |  | Matrix Codes |  |
| 10 Business Days For soil samples                |  | Commercial "A" (Level 1)  |  | NYASP Category A  |  | Matrix Codes |  |
| 5 Day RUSH                                       |  | Commercial "B" (Level 2)  |  | NYASP Category B  |  | Matrix Codes |  |
| 3 Day RUSH                                       |  | FULL T1 (Level 3-4)   |  | State Forms   |  | Matrix Codes |  |
| 2 Day RUSH                                       |  | NJ Reduced  |  | EOD Format  |  | Matrix Codes |  |
| 1 Day RUSH For aqueous samples                   |  | Commercial "C"  |  | Other   |  | Matrix Codes |  |
| Other  |  | NJ State of Health Quality Protocol Reporting   |  | Sample inventory is verified upon receipt in the Laboratory |  | Matrix Codes |  |
| Emergency & Rush T24 rates available via Lablink |  | Sample Chain of Custody must be documented below each time samples change possession, including courier delivery. |  | Sample inventory is verified upon receipt in the Laboratory |  | Matrix Codes |  |
| 1 5/11/16 1500                                   |  | 1 FedEx   |  | 2 5/11/16   |  | 2 5/11/16    |  |
| 3 5/11/16  |  | 3 5/11/16   |  | 4 5/11/16   |  | 4 5/11/16    |  |
| 5 5/11/16  |  | 5 5/11/16   |  | 6 5/11/16   |  | 6 5/11/16    |  |

MC45838A: Chain of Custody

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 ACCUTEST  
 MC45838A

## EXECUTIVE NARRATIVE

SDG No: **MC45838A** Laboratory: **Accutest, Massachusetts**  
Analysis: **MADEP VPH** Number of Samples: **1**  
Location: **BMSMC, Building 5 Area**  
**Humacao, PR**

**SUMMARY:** One (1) sample was analyzed for Volatiles TPHC Ranges by method MADEP VPH. Samples were validated following the METHOD FOR THE DETERMINATION OF VOLATILE PETROLEUM HYDROCARBONS (VPH) quality control criteria, Massachusetts Department of Environmental Protection, Revision 1.1 (2004). Also the general validation guidelines promulgated by the USEPA Hazardous Wastes Support Section. The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

Results are valid and can be used for decision making purposes.

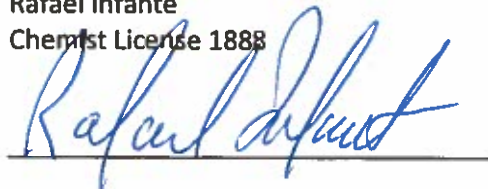
**Critical issues:** **None**  
**Major:** **None**  
**Minor:** **None**

**Critical findings:** **None**  
**Major findings:** **None**  
**Minor findings:** **1.** % differences in the rt5.5-7 hydrocarbon range did not meet the method and guidance document performance criteria in the initial calibration verification. No action taken, professional judgment.

**COMMENTS:** Results are valid and can be used for decision making purposes.

**Reviewers Name:** **Rafael Infante**  
**Chemist License 1888**

**Signature:**



**Date:** **June 13, 2016**

# SAMPLE ORGANIC DATA SAMPLE SUMMARY

Sample ID: MC45838-2

Sample location: BMSMC Building 5 Area

Sampling date: 5/10/2016

Matrix: Soil

## METHOD: MADEP VPH

| Analyte Name                 | Result | Units | Dilution | Factor | Lab Flag | Validation | Reportable |
|------------------------------|--------|-------|----------|--------|----------|------------|------------|
| Ç5 - C8 Aliphatics (Unadj.)  | 8100   | ug/kg | 1        |        | -        | U          | Yes        |
| Ç9 - C12 Aliphatics (Unadj.) | 8100   | ug/kg | 1        |        | -        | U          | Yes        |
| Ç9 - C10 Aromatics (Unadj.)  | 8100   | ug/kg | 1        |        | -        | U          | Yes        |
| Ç5 - C8 Aliphatics           | 8100   | ug/kg | 1        |        | -        | U          | Yes        |
| Ç9 - C12 Aliphatics          | 8100   | ug/kg | 1        |        | -        | U          | Yes        |

# DATA REVIEW WORKSHEETS

Type of validation Full: ☒ Limited: ☐ Project Number: MC45838A  
 Date: 05/10/2016  
 Shipping date: 05/10/2016  
 EPA Region: 2

## REVIEW OF VOLATILE PETROLEUM HYDROCARBON (VPHs) PACKAGE

The following guidelines for evaluating volatile organics were created to delineate required validation actions. This document will assist the reviewer in using professional judgment to make more informed decision and in better serving the needs of the data users. The sample results were assessed according to the data validation guidance documents in the following order of precedence METHOD FOR THE DETERMINATION OF VOLATILE PETROLEUM HYDROCARBONS (VPH), Massachusetts Department of Environmental Protection, Revision 1.1 (2004). Also the general validation guidelines promulgated by the USEPA Hazardous Wastes Support Section. The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

The hardcopied (laboratory name) Accutest Laboratories data package received has been reviewed and the quality control and performance data summarized. The data review for SVOCs included:

Lab. Project/SDG No.: MC45838A Sample matrix: Soil  
 No. of Samples: 1  
 Field blank No.:  
 Equipment blank No.:  
 Trip blank No.:  
 Field duplicate No.:

|   |   |
|---|---|
| <input checked="" type="checkbox"/> Data Completeness                   | <input checked="" type="checkbox"/> Laboratory Control Spikes |
| <input checked="" type="checkbox"/> Holding Times                       | <input checked="" type="checkbox"/> Field Duplicates          |
| <input type="checkbox"/> GC/MS Tuning                                   | <input checked="" type="checkbox"/> Calibrations              |
| <input type="checkbox"/> Internal Standard Performance                  | <input checked="" type="checkbox"/> Compound Identifications  |
| <input checked="" type="checkbox"/> Blanks                              | <input checked="" type="checkbox"/> Compound Quantitation     |
| <input checked="" type="checkbox"/> Surrogate Recoveries                | <input checked="" type="checkbox"/> Quantitation Limits       |
| <input checked="" type="checkbox"/> Matrix Spike/Matrix Spike Duplicate |   |

Overall Comments: Volatiles by GC by Method MADEP\_VPH\_REV\_1.1.  
 (C5\_to\_C12\_Aliphatics; C9\_to\_C10\_Aromatics)

### Definition of Qualifiers:

J- Estimated results  
 U- Compound not detected  
 R- Rejected data  
 UJ- Estimated nondetected

Reviewer:   
 Date: 06/13/2016

## DATA REVIEW WORKSHEETS

All criteria were met   x    
Criteria were not met and/or see below           

## I. DATA COMPLETNESS

A. **Data Package:**

### MISSING INFORMATION

DATE LAB. CONTACTEDDATE RECEIVED[illegible]

B. Other

**Discrepancies:**

This image shows a single sheet of white paper with horizontal ruling lines. The lines are evenly spaced and run across the width of the page. There are no margins or other markings visible.



## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met and/or see below           

### HOLDING TIMES

The objective of this parameter is to ascertain the validity of the results based on the holding time of the sample from time of collection to the time of extraction, and subsequently from the time of extraction to the time of analysis.

Complete table for all samples and note the analysis and/or preservation not within criteria

| SAMPLE ID   | DATE SAMPLED | DATE EXTRACTED | DATE ANALYZED | ACTION |
|---|--------------|----------------|---------------|--------|
|   |              |                |               |        |
|   |              |                |               |        |
|   |              |                |               |        |
| Samples analyzed within method recommended holding time |              |                |               |        |
|   |              |                |               |        |
|   |              |                |               |        |
|   |              |                |               |        |

### Criteria

#### Preservation:

Samples analyzed with ambient purge temperature: Samples must be acidified to a pH of 2.0 or less at the time of collection.

Samples analyzed with heated purge temperature: Samples must be treated to a pH of 11.0 or greater at the time of collection.

Methanol preservation of soil/sediment samples is mandatory. Methanol (purge-and-trap grade) must be added to the sample vial before or immediately after sample collection. In lieu of the in-field preservation of samples with methanol, soil samples may be obtained in specially-designed air tight sampling devices, provided that the samples are extruded and preserved in methanol within 48 hours of collection.

#### Holding times:

Aqueous samples using ambient or heated purge - analyze within 14 days.

Soil/sediment samples - analysis within 28 days.

Cooler temperature (Criteria:  $4 \pm 2$  °C):   4.8°C  

#### Actions: Qualify positive results/nondetects as follows:

If holding times are exceeded, estimate positive results (J) and nondetects (UJ).

If holding times are grossly exceeded, use professional judgment to qualify data. The data reviewer may choose to estimate positive results (J) and rejects nondetects (R).

If samples were not at the proper temperature ( $> 10^{\circ}\text{C}$ ) or improperly preserved, use professional judgment to qualify the results.

## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met and/or see below           

### CALIBRATIONS VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration:   01/12/16  

Dates of initial calibration verification:   01/12/16  

Instrument ID numbers:           GCAB          

Matrix/Level:           AQUEOUS/MEDIUM          

| DATE     | LAB FILE ID# | ANALYTE | CRITERIA OUT<br>RFs, %RSD, %D, r | SAMPLES<br>AFFECTED |
|----------|--------------|---------|----------------------------------|---------------------|
| GCAB     |              |         |                                  |                     |
| 01/12/16 | icv-5058-50  | rt5.5-7 | 22.6                             | None                |
|          |              |         |                                  |                     |
|          |              |         |                                  |                     |
|          |              |         |                                  |                     |
|          |              |         |                                  |                     |

**Note:** Initial and initial calibration verification meet method specific requirements except in the cases described in this document. No action taken, professional judgment.

#### Criteria- ICAL

- Five point calibration curve.
- The percent relative standard deviation (%RSD) of the calibration factor must be equal to or less than 25% over the working range for the analyte of interest. When this condition is met, linearity through the origin may be assumed, and the average calibration factor is used in lieu of a calibration curve.
- A collective calibration factor must also be established for each hydrocarbon range of interest. Calculate the collective CFs for C5-C8 Aliphatic Hydrocarbons and C9-C12 Aliphatic Hydrocarbons using the FID chromatogram. Calculate the collective CF for the C9-C10 Aromatic Hydrocarbons using the PID chromatogram. Tabulate the summation of the peak areas of all components in that fraction against the total concentration injected. The %RSD of the calibration factor must be equal to or less than 25% over the working range for the hydrocarbon range of interest.

## DATA REVIEW WORKSHEETS

### Criteria- CCAL

- At a minimum, the working calibration factor must be verified on each working day, after every 20 samples, and at the end of the analytical sequence by the injection of a mid-level continuing calibration standard to verify instrument performance and linearity.
- If the percent difference (%D) for any analyte varies from the predicted response by more than  $\pm 25\%$ , a new five-point calibration must be performed for that analyte. Greater percent differences are permissible for n-nonane. If the %D for n-nonane is greater than 30, note the nonconformance in the case narrative. It should be noted that the %Ds are calculated when CFs are used for the initial calibration and percent drifts are calculated when calibration curves using linear regression are used for the initial calibration.

### Actions:

If %RSD > 25% for target compounds or a correlation coefficient < 0.99, estimate positive results (J) and use professional judgment to qualify nondetects.

If % D > 25% (> 30 for nonane), estimate positive results (J) and nondetects (UJ).

### CALIBRATIONS VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration: \_\_\_\_\_ 01/12/16 \_\_\_\_\_

Dates of continuing calibration verification: \_\_\_\_\_ 05/12/16 \_\_\_\_\_

Dates of final calibration verification: \_\_\_\_\_ 05/12/16 \_\_\_\_\_

Instrument ID numbers: \_\_\_\_\_ GCAB \_\_\_\_\_

Matrix/Level: \_\_\_\_\_ AQUEOUS/MEDIUM \_\_\_\_\_

| DATE | LAB FILE ID# | ANALYTE | CRITERIA OUT<br>RFs, %RSD, %D, r | SAMPLES<br>AFFECTED |
|------|--------------|---------|----------------------------------|---------------------|
|      |              |         |                                  |                     |
|      |              |         |                                  |                     |
|      |              |         |                                  |                     |
|      |              |         |                                  |                     |
|      |              |         |                                  |                     |
|      |              |         |                                  |                     |
|      |              |         |                                  |                     |
|      |              |         |                                  |                     |

**Note:** Continuing and final calibration verification meet method specific requirements.

A separate worksheet should be filled for each initial curve

# DATA REVIEW WORKSHEETS

All criteria were met   X    
Criteria were not met and/or see below           

## V A. BLANK ANALYSIS RESULTS (Sections 1 & 2)

The assessment of the blank analysis results is to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks apply only to blanks associated with the samples, including trip, equipment, and laboratory blanks. If problems with any blanks exist, all data associated with the case must be carefully evaluated to determine whether or not there is an inherent variability in the data for the case, or if the problem is an isolated occurrence not affecting other data. A Laboratory Method Blank must be run after samples suspected of being highly contaminated to determine if sample carryover has occurred.

List the contamination in the blanks below. High and low levels blanks must be treated separately.

### Laboratory blanks

| DATE ANALYZED | LAB ID | LEVEL/ MATRIX | COMPOUND | CONCENTRATION UNITS |
|---------------|--------|---------------|----------|---------------------|
|---------------|--------|---------------|----------|---------------------|

  METHOD BLANKS MEET THE METHOD SPECIFIC CRITERIA  

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

### Field/Trip/Equipment

A methanol trip blank or acidified reagent water trip blank **should** continually accompany each soil/sediment sample or water sample batch, respectively, during sampling, storage, and analysis.

| DATE ANALYZED | LAB ID | LEVEL/ MATRIX | COMPOUND | CONCENTRATION UNITS |
|---------------|--------|---------------|----------|---------------------|
|---------------|--------|---------------|----------|---------------------|

  NO TRIP/FIELD/EQUIPMENT BLANKS ASSOCIATED WITH THIS DATA PACKAGE  

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

## DATA REVIEW WORKSHEETS

All criteria were met   X    
Criteria were not met and/or see below           

### V      B.      BLANK ANALYSIS RESULTS (Section 3)

#### Blank Actions

The ALs for samples which have been diluted should be corrected for the sample dilution factor and/or % moisture, where applicable. Peaks must not be detected above the Reporting Limit within the retention time window of any analyte of interest. The hydrocarbon ranges must not be detected at a concentration greater than 10% of the most stringent MCP cleanup standard. Specific actions area as follows:

If the concentration is < sample quantitation limit (SQL) and < AL, report the compound as not detected (U) at the SQL.

If the concentration is  $\geq$  SQL but < AL, report the compound as not detected (U) at the reported concentration.

If the concentration is > AL, report the concentration unqualified.

## DATA REVIEW WORKSHEETS

All criteria were met   X    
Criteria were not met and/or see below           

### SURROGATE SPIKE RECOVERIES

Laboratory performance of individual samples is established by evaluation of surrogate spike recoveries. All samples are spiked with surrogate compounds prior to sample analysis. The accuracy of the analysis is measured by the surrogate percent recovery. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the validation of data is frequently subjective and demands analytical experience and professional judgment.

List the percent recoveries (%Rs) which do not meet the criteria for surrogate recovery.

Matrix: solid/aqueous

| SAMPLE ID  | SURROGATE COMPOUND     | ACTION |
|--|------------------------|--------|
|  | 2,3,4-Trifluorotoluene |        |
| <u>  SURROGATE_STANDARD_RECOVERIES_WITHIN_LABORATORY_CONTROL  </u> |                        |        |
| <u>  LIMITS  </u>  |                        |        |
|  |                        |        |
|  |                        |        |
|  |                        |        |
|  |                        |        |
|  |                        |        |

QC Limits\* (Aqueous)

       LL   to   UL          70   to  130         to               to       

QC Limits\* (Solid)

       LL   to   UL               to               to               to       

It is recommended that surrogate standard recoveries be monitored and documented on a continuing basis. At a minimum, when surrogate recovery from a sample, blank, or QC sample is less than 70% or more than 130%, check calculations to locate possible errors, check the fortifying standard solution for degradation, and check changes in instrument performance.

- If the cause cannot be determined, reanalyze the sample unless one of the following exceptions applies:
- (1) Obvious interference is present on the chromatogram (e.g., unresolved complex mixture).
  - (2) Percent moisture of associated soil/sediment sample is >25% and surrogate recovery is >10%; or
  - (3) The surrogate exhibits high recovery and associated target analytes or hydrocarbon ranges are not detected in sample.

If a sample with a surrogate recovery outside of the acceptable range is not reanalyzed based on any of these aforementioned exceptions, this information must be noted on the data report form and discussed in the Executive Report. Analysis of the sample on dilution may diminish matrix-related surrogate recovery problems. This approach can be used as long as the reporting limits to evaluate applicable MCP standards can still be achieved with the dilution. If not, reanalysis without dilution must be performed.

## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met and/or see below           

### VII. A MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

This data is generated to determine long term precision and accuracy in the analytical method for various matrices. This data alone cannot be used to evaluate the precision and accuracy of individual samples.

At the request of the data user, and in consideration of sample matrices and data quality objectives, matrix spikes and matrix duplicates may be analyzed with every batch of 20 samples or less per matrix.

- **Matrix duplicate** - Matrix duplicates are prepared by analyzing one sample in duplicate. The purpose of the matrix duplicates is to determine the homogeneity of the sample matrix as well as analytical precision. The RPD of detected results in the matrix duplicate samples must not exceed 50 when the results are greater than 5x the reporting limit.
- The desired spiking level is 50% of the highest calibration standard. However, the total concentration in the MS (including the MS and native concentration in the unspiked sample) should not exceed 75% of the highest calibration standard in order for a proper evaluation to be performed. The purpose of the matrix spike is to determine whether the sample matrix contributes bias to the analytical results. The corrected concentrations of each analyte within the matrix spiking solution must be within 70 - 130% of the true value. Lower recoveries of n-nonane are permissible (if included in the calibration of the C9-C12 aliphatic range), but must be noted in the narrative if <30%.

#### MS/MSD Recoveries and Precision Criteria

Sample ID: MC45816-2\_MS/MSD Matrix/Level: Soil

List the %Rs, RPD of the compounds which do not meet the QC criteria.

| MS OR MSD  | COMPOUND | % R | RPD | QC LIMITS | ACTION |
|--|----------|-----|-----|-----------|--------|
| <u>_MS/MSD_recoveries_and_RPD_within_laboratory_control_limits._</u> |          |     |     |           |        |
| <u> </u>   |          |     |     |           |        |
| <u> </u>   |          |     |     |           |        |
| <u> </u>   |          |     |     |           |        |

## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met and/or see below           

No action is taken on MS/MSD results alone to qualify the entire case. However, used informed professional judgment, the data reviewer may use the MS/MSD results in conjunction with other QC criteria and determine the need for some qualification of the data. In those instances where it can be determined that the results of the MS/MSD affect only the sample spiked, the qualification should be limited to this sample alone. However, it may be determined through the MS/MSD results that the laboratory is having a systematic problem in the analysis of one or more analytes, which affects the associated samples.

### 2. MS/MSD – Unspiked Compounds

List the concentrations of the unspiked compounds and determine the % RSDs of these compounds in the unspiked sample, matrix spike, and matrix spike duplicate.

| COMPOUND | CONCENTRATION |    | MSD | %RPD | ACTION |
|----------|---------------|----|-----|------|--------|
|          | SAMPLE        | MS |     |      |        |
|          |               |    |     |      |        |
|          |               |    |     |      |        |
|          |               |    |     |      |        |
|          |               |    |     |      |        |
|          |               |    |     |      |        |
|          |               |    |     |      |        |
|          |               |    |     |      |        |

Criteria: None specified, use  $\%RSD \leq 50$  as professional judgment.

Actions:

If the  $\% RSD > 50$ , qualify the results in the spiked sample as estimate (J).

If the  $\% RSD$  is not calculable (NC) due to nondetect value in the sample, MS, and/or MSD, use professional judgment to qualify sample data.

A separate worksheet should be used for each MS/MSD pair.



## DATA REVIEW WORKSHEETS

All criteria were met   X    
Criteria were not met and/or see below           

### VIII. LABORATORY CONTROL SAMPLE (LCS/LCSD) ANALYSIS

This data is generated to determine accuracy of the analytical method for various matrices.

#### 1. LCS Recoveries Criteria

List the %R of compounds which do not meet the criteria

| LCS ID   | COMPOUND | % R | QC LIMIT | ACTION |
|--|----------|-----|----------|--------|
| <u>  LCS_RECOVERY_WITHIN_LABORATORY_CONTROL_LIMITS  </u> |          |     |          |        |
|  |          |     |          |        |
|  |          |     |          |        |
|  |          |     |          |        |

#### Criteria:

- \* Refer to QAPP for specific criteria.
- \* The spike recovery must be between 70% and 130%. Lower recoveries of n-nonane are permissible (if included in the calibration of the C9-C12 aliphatic range). If the recovery of n-nonane is <30%, note the nonconformance in the executive narrative.

#### Actions:

Actions on LCS recovery should be based on both the number of compounds that are outside the %R criteria and the magnitude of the exceedance of the criteria.

If the %R of the analyte is > UL, qualify all positive results (j) for the affected analyte in the associated samples and accept nondetects.

If the %R of the analyte is < LL, qualify all positive results (j) and reject (R) nondetects for the affected analyte in the associated samples.

If more than half the compounds in the LCS are not within the required recovery criteria, qualify all positive results as (J) and reject nondetects (R) for all target analyte(s) in the associated samples.

#### 2. Frequency Criteria:

Where LCS analyzed at the required frequency and for each matrix (1 per 20 samples per matrix)? Yes or No.

If no, the data may be affected. Use professional judgment to determine the severity of the effect and qualify data accordingly. Discuss any actions below and list the samples affected. Discuss the actions below:

## DATA REVIEW WORKSHEETS

All criteria were met N/A  
Criteria were not met and/or see below \_\_\_\_\_

### IX. FIELD/LABORATORY DUPLICATE PRECISION

Sample IDs: \_\_\_\_\_ - \_\_\_\_\_

Matrix: \_\_\_\_\_ - \_\_\_\_\_

Field/laboratory duplicates samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than laboratory duplicates which measures only laboratory performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field duplicate samples.

| COMPOUND  | SQL | SAMPLE CONC. | DUPLICATE CONC. | RPD | ACTION |
|---|-----|--------------|-----------------|-----|--------|
|   |     |              |                 |     |        |
|   |     |              |                 |     |        |
| No field/laboratory duplicate analyzed with this data package. MS/MSD recoveries RPD used to assess precision. RPD within laboratory and generally acceptable control limits. |     |              |                 |     |        |
|   |     |              |                 |     |        |
|   |     |              |                 |     |        |

#### Criteria:

The project QAPP should be reviewed for project-specific information.  
RPD  $\pm$  30% for aqueous samples, RPD  $\pm$  50 % for solid samples if results are  $\geq$  SQL.  
If both samples and duplicate are  $< 5$  SQL, the RPD criteria is doubled.

SQL = soil quantitation limit

#### Actions:

If both the sample and the duplicate results are nondetects (ND), the RPD is not calculable (NC). No action is needed.

Qualify as estimated positive results (J) and nondetects (UJ) for the compound that exceeded the above criteria.

If one sample result is not detected and the other is  $\geq 5x$  the SQL qualify (J/UJ).

**Note:** If SQLs for the sample and duplicate are significantly different, use professional judgment to determine if qualification is appropriate.

If one sample value is not detected and the other is  $< 5x$  the SQL, use professional judgment to determine if qualification is appropriate.

## DATA REVIEW WORKSHEETS

All criteria were met   X    
Criteria were not met and/or see below       

### XI. COMPOUND IDENTIFICATION

The compound identification evaluation is to verify that the laboratory correctly identified target analytes as well as tentatively identified compounds (TICs).

1. Verify that the target analytes were within the retention time windows.
  - Retention time windows must be re-established for each Target VPH Analyte each time a new GC column is installed, and must be verified and/or adjusted on a daily basis.
  - Coelution of the m- and p- xylene isomers is permissible.
  - All surrogates must be adequately resolved from individual Target Analytes included in the VPH Component Standard.
  - For the purposes of this method, adequate resolution is assumed to be achieved if the height of the valley between two peaks is less than 25% of the average height of the two peaks.
  - The n-pentane (C5) and MtBE peaks must be adequately resolved from any solvent front that may be present on the FID and PID chromatograms, respectively.

**Note:** Target analytes were within the retention time window.

2. If target analytes and/or TICs were not correctly identified, request that the laboratory resubmit the corrected data.

## DATA REVIEW WORKSHEETS

All criteria were met   X    
Criteria were not met and/or see below           

### XII. QUANTITATION LIMITS AND SAMPLE RESULTS

The sample quantitation evaluation is to verify laboratory quantitation results.

- In the space below, please show a minimum of one sample calculation:

MC45838-2                      2,3,4-trifluorotoluene                      RF =  $4.100 \times 10^5$

FID

$$[\ ] = (18536704)/(4.015 \times 10^5)$$

$$[\ ] = 45.21 \text{ ppb} \quad \text{Ok}$$

MC45838-2                      Fluorobenzene                      RF =  $4.401 \times 10^5$

PID

$$[\ ] = (21945897)/(4.401 \times 10^5)$$

$$[\ ] = 49.87 \text{ ppb} \quad \text{Ok}$$

- If requested, verify that the results were above the laboratory method detection limit (MDLs).

- If dilutions performed, were the SQLs elevated accordingly by the laboratory? List the affected samples and dilution factor in the table below.

| SAMPLE ID | DILUTION FACTOR | REASON FOR DILUTION |
|-----------|-----------------|---------------------|
|           |                 |                     |
|           |                 |                     |
|           |                 |                     |
|           |                 |                     |
|           |                 |                     |
|           |                 |                     |
|           |                 |                     |
|           |                 |                     |
|           |                 |                     |

If dilution was not performed and the results were above the concentration range, estimate results (J) for the affected compounds. List the affected samples/compounds:

\_\_\_\_\_

\_\_\_\_\_

## EXECUTIVE NARRATIVE

SDG No: **MC45838A** Laboratory: **Accutest, Massachusetts**  
Analysis: **MADEP EPH** Number of Samples: **1**  
Location: **BMSMC, Building 5 Area**  
**Humacao, PR**

**SUMMARY:** One (1) sample was analyzed for Extractable TPHC Ranges by method MADEP EPH. Samples were validated following the METHOD FOR THE DETERMINATION OF EXTRACTABLE PETROLEUM HYDROCARBONS (EPH) quality control criteria, Massachusetts Department of Environmental Protection, Revision 1.1 (2004). Also the general validation guidelines promulgated by the USEPA Hazardous Wastes Support Section. The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

Results are valid and can be used for decision making purposes.

**Critical issues:** **None**  
**Major:** **None**  
**Minor:** **None**

**Critical findings:** **None**  
**Major findings:** **None**  
**Minor findings:** **1. MS/MSD % recoveries and RPD outside the laboratory control limits. MS/MSD results and RPD apply to unspiked sample. Unspiked sample was from another project. No action taken.**

**COMMENTS:** Results are valid and can be used for decision making purposes.

**Reviewers Name:** **Rafael Infante**  
**Chemist License 1888**

**Signature:**



**Date:** **Juan 13, 2016**

# **SAMPLE ORGANIC DATA SAMPLE SUMMARY**

**Sample ID: MC45838-2**

**Sample location: BMSMC Building 5 Area**

**Sampling date: 5/10/2016**

**Matrix: Soil**

## **METHOD: MADEP EPH**

| <b>Analyte Name</b>                 | <b>Result</b> | <b>Units</b> | <b>Dilution Factor</b> | <b>Lab Flag</b> | <b>Validation</b> | <b>Reportable</b> |
|-------------------------------------|---------------|--------------|------------------------|-----------------|-------------------|-------------------|
| <b>Ç11 - C22 Aromatics (Unadj.)</b> | <b>24000</b>  | <b>ug/kg</b> | <b>1</b>               | <b>-</b>        | <b>U</b>          | <b>Yes</b>        |
| <b>Ç9 - C18 Aliphatics</b>          | <b>12000</b>  | <b>ug/kg</b> | <b>1</b>               | <b>-</b>        | <b>U</b>          | <b>Yes</b>        |
| <b>Ç19 - C36 Aliphatics</b>         | <b>12000</b>  | <b>ug/kg</b> | <b>1</b>               | <b>-</b>        | <b>U</b>          | <b>Yes</b>        |
| <b>Ç11 - C22 Aromatics</b>          | <b>24000</b>  | <b>ug/kg</b> | <b>1</b>               | <b>-</b>        | <b>U</b>          | <b>Yes</b>        |

# DATA REVIEW WORKSHEETS

Type of validation Full: ☒ Limited: ☐ Project Number: MC45838A Date: 05/10/2016 Shipping date: 05/10/2016 EPA Region: 2

## REVIEW OF EXTRACTABLE PETROLEUM HYDROCARBON (EPHs) PACKAGE

The following guidelines for evaluating volatile organics were created to delineate required validation actions. This document will assist the reviewer in using professional judgment to make more informed decision and in better serving the needs of the data users. The sample results were assessed according to the data validation guidance documents in the following order of precedence METHOD FOR THE DETERMINATION OF EXTRACTABLE PETROLEUM HYDROCARBONS (VPH), Massachusetts Department of Environmental Protection, Revision 1.1 (2004). Also the general validation guidelines promulgated by the USEPA Hazardous Wastes Support Section. The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

The hardcopied (laboratory name) Accutest Laboratories data package received has been reviewed and the quality control and performance data summarized. The data review for SVOCs included:

Lab. Project/SDG No.: MC45838A Sample matrix: Soil  
No. of Samples: 1  
Field blank No.: -  
Equipment blank No.: -  
Trip blank No.: -  
Field duplicate No.: -

|   |   |
|---|---|
| <input checked="" type="checkbox"/> Data Completeness                   | <input checked="" type="checkbox"/> Laboratory Control Spikes |
| <input checked="" type="checkbox"/> Holding Times                       | <input checked="" type="checkbox"/> Field Duplicates          |
| <input type="checkbox"/> GC/MS Tuning                                   | <input checked="" type="checkbox"/> Calibrations              |
| <input type="checkbox"/> Internal Standard Performance                  | <input checked="" type="checkbox"/> Compound Identifications  |
| <input checked="" type="checkbox"/> Blanks                              | <input checked="" type="checkbox"/> Compound Quantitation     |
| <input checked="" type="checkbox"/> Surrogate Recoveries                | <input checked="" type="checkbox"/> Quantitation Limits       |
| <input checked="" type="checkbox"/> Matrix Spike/Matrix Spike Duplicate |   |

Overall Comments:  
Extractable Petroleum Hydrocarbons by GC by Method MADEP\_EPH\_REV\_1.1.  
(C9\_to\_C36\_Aliphatics; C11\_to\_C22\_Aromatics)

### Definition of Qualifiers:

J- Estimated results  
U- Compound not detected  
R- Rejected data  
UJ- Estimated non-detect

Reviewer:   
Date: 06/13/2016

## DATA REVIEW WORKSHEETS

All criteria were met   x    
Criteria were not met and/or see below           

### I. DATA COMPLETNESS

#### A. Data Package:

MISSING INFORMATION

DATE LAB. CONTACTED

DATE RECEIVED

|  |  |  |
|--|--|--|
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#### B. Other

Discrepancies:

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## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met and/or see below           

### HOLDING TIMES

The objective of this parameter is to ascertain the validity of the results based on the holding time of the sample from time of collection to the time of extraction, and subsequently from the time of extraction to the time of analysis.

Complete table for all samples and note the analysis and/or preservation not within criteria

| SAMPLE ID   | DATE SAMPLED | DATE EXTRACTED | DATE ANALYZED | ACTION |
|---|--------------|----------------|---------------|--------|
|   |              |                |               |        |
|   |              |                |               |        |
|   |              |                |               |        |
| Samples extracted and analyzed within method recommended holding time |              |                |               |        |
|   |              |                |               |        |
|   |              |                |               |        |
|   |              |                |               |        |

### Criteria

#### Preservation:

Aqueous samples must be acidified to a pH of 2.0 or less at the time of collection.  
 Soil samples must be cooled at  $4 \pm 2$  °C immediately after collection.

#### Holding times:

Samples must be extracted within 14 days of collection, and analyzed within 40 days of extraction.

Cooler temperature (Criteria:  $4 \pm 2$  °C):   4.8°C  

### Actions: Qualify positive results/nondetects as follows:

If holding times are exceeded, estimate positive results (J) and nondetects (UJ).

If holding times are grossly exceeded, use professional judgment to qualify data. The data reviewer may choose to estimate positive results (J) and rejects nondetects (R).

If samples were not at the proper temperature ( $> 10^{\circ}\text{C}$ ) or improperly preserved, use professional judgment to qualify the results.

All criteria were met   X    
 Criteria were not met and/or see below

## DATA REVIEW WORKSHEETS

### CALIBRATIONS VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration: \_\_\_\_\_ 02/04/16 \_\_\_\_\_

Dates of initial calibration verification: \_\_\_\_\_ 02/04/13 \_\_\_\_\_

Instrument ID numbers: \_\_\_\_\_ GCDE \_\_\_\_\_

Matrix/Level: \_\_\_\_\_ AQUEOUS/MEDIUM \_\_\_\_\_

| DATE  | LAB FILE ID# | ANALYTE | CRITERIA OUT<br>RFs, %RSD, %D, r | SAMPLES<br>AFFECTED |
|---|--------------|---------|----------------------------------|---------------------|
|   |              |         |                                  |                     |
| Initial calibration and initial calibration verification meet method specific requirements. |              |         |                                  |                     |
|   |              |         |                                  |                     |

#### Criteria- ICAL

- Five point calibration curve.
- The percent relative standard deviation (%RSD) of the calibration factor must be equal to or less than 25% over the working range for the analyte of interest. When this condition is met, linearity through the origin may be assumed, and the average calibration factor is used in lieu of a calibration curve.
- A collective calibration factor must also be established for each hydrocarbon range of interest. Calculate the collective CFs for C9-C18 Aliphatic Hydrocarbons, C19-C36 Aliphatic Hydrocarbons, and C11-C22 Aromatic Hydrocarbons using the FID chromatogram. Tabulate the summation of the peak areas of all components in that fraction against the total concentration injected. The %RSD of the calibration factor must be equal to or less than 25% over the working range for the hydrocarbon range of interest.
  - The area for the surrogates must be subtracted from the area summation of the range in which they elute.
  - The areas associated with naphthalene and 2-methylnaphthalene in the aliphatic range standard must be subtracted from the uncorrected collective C9-C18 Aliphatic Hydrocarbon range area prior to calculating the CF.

#### Criteria- CCAL

- At a minimum, the working calibration factor must be verified on each working day, after every 20 samples or every 24 hours (whichever is more frequent), and at the

## DATA REVIEW WORKSHEETS

end of the analytical sequence by the injection of a mid-level continuing calibration standard to verify instrument performance and linearity.

- If the percent difference (%D) for any analyte varies from the predicted response by more than  $\pm 25\%$ , a new five-point calibration must be performed for that analyte. Greater percent differences are permissible for n-nonane. If the %D for n-nonane is greater than 30, note the nonconformance in the case narrative. It should be noted that the %Ds are calculated when CFs are used for the initial calibration and percent drifts are calculated when calibration curves using linear regression are used for the initial calibration.

### Actions:

If %RSD > 25% for target compounds or a correlation coefficient < 0.99, estimate positive results (J) and use professional judgment to qualify nondetects.

If % D > 25% (> 30 for nonane), estimate positive results (J) and nondetects (UJ).

### CALIBRATIONS VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration: \_\_\_\_\_ 02/04/16 \_\_\_\_\_

Dates of continuing calibration verification: \_\_\_\_\_ 05/31/16 \_\_\_\_\_

Dates of final calibration verification: \_\_\_\_\_ 05/31/16 \_\_\_\_\_

Instrument ID numbers: \_\_\_\_\_ GCDE \_\_\_\_\_

Matrix/Level: \_SOIL/AQUEOUS/MEDIUM\_\_\_\_\_

| DATE  | LAB FILE ID# | ANALYTE | CRITERIA OUT<br>RFs, %RSD, %D, r | SAMPLES<br>AFFECTED |
|---|--------------|---------|----------------------------------|---------------------|
|   |              |         |                                  |                     |
| Continuing and ending calibration verification meet method specific requirements. |              |         |                                  |                     |
|   |              |         |                                  |                     |

A separate worksheet should be filled for each initial curve

All criteria were met   X    
Criteria were not met and/or see below \_\_\_\_\_

V A. BLANK ANALYSIS RESULTS (Sections 1 & 2)

## DATA REVIEW WORKSHEETS

The assessment of the blank analysis results is to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks apply only to blanks associated with the samples, including trip, equipment, and laboratory blanks. If problems with any blanks exist, all data associated with the case must be carefully evaluated to determine whether or not there is an inherent variability in the data for the case, or if the problem is an isolated occurrence not affecting other data. A Laboratory Method Blank must be run after samples suspected of being highly contaminated to determine if sample carryover has occurred.

List the contamination in the blanks below. High and low levels blanks must be treated separately.

### Laboratory blanks

| DATE<br>ANALYZED | LAB ID | LEVEL/<br>MATRIX | COMPOUND | CONCENTRATION<br>UNITS |
|------------------|--------|------------------|----------|------------------------|
|------------------|--------|------------------|----------|------------------------|

\_\_\_\_METHOD BLANKS MEET THE METHOD SPECIFIC CRITERIA\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

### Field/Trip/Equipment

| DATE<br>ANALYZED | LAB ID | LEVEL/<br>MATRIX | COMPOUND | CONCENTRATION<br>UNITS |
|------------------|--------|------------------|----------|------------------------|
|------------------|--------|------------------|----------|------------------------|

\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
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\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

\_\_\_\_NO TRIP/FIELD/EQUIPMENT BLANKS ANALYZED ASSOCIATED WITH THIS\_\_\_\_DA  
TA PACKAGE.\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

All criteria were met \_\_X\_\_  
Criteria were not met and/or see below \_\_\_\_\_

## DATA REVIEW WORKSHEETS

### Blank Actions

The ALs for samples which have been diluted should be corrected for the sample dilution factor and/or % moisture, where applicable. Peaks must not be detected above the Reporting Limit within the retention time window of any analyte of interest. The hydrocarbon ranges must not be detected at a concentration greater than 10% of the most stringent MCP cleanup standard. Specific actions area as follows:

If the concentration is < sample quantitation limit (SQL) and < AL, report the compound as not detected (U) at the SQL.

If the concentration is  $\geq$  SQL but < AL, report the compound as not detected (U) at the reported concentration.

If the concentration is > AL, report the concentration unqualified.

All criteria were met   X  

Criteria were not met and/or see below           

### SURROGATE SPIKE RECOVERIES

## DATA REVIEW WORKSHEETS

Laboratory performance of individual samples is established by evaluation of surrogate spike recoveries. All samples are spiked with surrogate compounds prior to sample analysis. The accuracy of the analysis is measured by the surrogate percent recovery. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the validation of data is frequently subjective and demands analytical experience and professional judgment.

List the percent recoveries (%Rs) which do not meet the criteria for surrogate recovery.

Matrix: solid/aqueous

| SAMPLE ID   | SURROGATE COMPOUND |    |    |    | ACTION |
|---|--------------------|----|----|----|--------|
|   | S1                 | S2 | S3 | S4 |        |
| SURROGATE STANDARDS RECOVERIES WITHIN LABORATORY CONTROL LIMITS |                    |    |    |    | SU     |
|   |                    |    |    |    |        |
|   |                    |    |    |    |        |
|   |                    |    |    |    |        |
|   |                    |    |    |    |        |

S1 = o-Terphenyl 40-140%

S2 = 2-Fluorobiphenyl 40-140%

S3 = 1-Chlorooctadecane 40-140%

S4 = 2-Bromonaphthalene 40-140%

QC Limits (%)\* (Aqueous/Solid)

\_LL\_ to \_UL\_    \_40\_ to \_140\_    \_40\_ to \_140\_    \_40\_ to \_140\_    \_40\_ to \_140\_

QC Limits\* (Solid)

\_LL\_ to \_UL\_    \_\_\_ to \_\_\_    \_\_\_ to \_\_\_    \_\_\_ to \_\_\_    \_\_\_ to \_\_\_

It is recommended that surrogate standard recoveries be monitored and documented on a continuing basis. At a minimum, when surrogate recovery from a sample, blank, or QC sample is less than 40% or more than 140%, check calculations to locate possible errors, check the fortifying standard solution for degradation, and check changes in instrument performance.

If the cause cannot be determined, reanalyze the sample unless one of the following exceptions applies:

- (1) Obvious interference is present on the chromatogram (e.g., unresolved complex mixture);
- (2) The surrogate exhibits high recovery and associated target analytes or hydrocarbon ranges are not detected in sample.

If a sample with a surrogate recovery outside of the acceptable range is not reanalyzed based on any of these aforementioned exceptions, this information must be noted on the data report form and discussed in the Executive Report. Analysis of the sample on dilution may diminish matrix-related surrogate recovery problems. This approach can be used as long as the reporting limits to evaluate applicable MCP standards can still be achieved with the dilution. If not, reanalysis without dilution must be performed.

# DATA REVIEW WORKSHEETS

All criteria were met \_\_\_\_\_  
Criteria were not met and/or see below X

## VII. A MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

This data is generated to determine long term precision and accuracy in the analytical method for various matrices. This data alone cannot be used to evaluate the precision and accuracy of individual samples.

At the request of the data user, and in consideration of sample matrices and data quality objectives, matrix spikes and matrix duplicates may be analyzed with every batch of 20 samples or less per matrix.

- **Matrix duplicate** - Matrix duplicates are prepared by analyzing one sample in duplicate. The purpose of the matrix duplicates is to determine the homogeneity of the sample matrix as well as analytical precision. The RPD of detected results in the matrix duplicate samples must not exceed 50 when the results are greater than 5x the reporting limit.
- The desired spiking level is 50% of the highest calibration standard. However, the total concentration in the MS (including the MS and native concentration in the unspiked sample) should not exceed 75% of the highest calibration standard in order for a proper evaluation to be performed. The purpose of the matrix spike is to determine whether the sample matrix contributes bias to the analytical results. The corrected concentrations of each analyte within the matrix spiking solution must be within 40 - 140% of the true value. Lower recoveries of n-nonane are permissible but must be noted in the narrative if <30%.

### MS/MSD Recoveries and Precision Criteria

Sample ID: FA33755-1R Matrix/Level: Soil

List the %Rs, RPD of the compounds which do not meet the QC criteria.

The QC reported here applies to the following samples:  
**MC45838-2**

Method: **MADEP EPH REV 1.1**

| Compound                      | FA33755-1R<br>ug/kg | Q | Spike<br>ug/kg | MS<br>ug/kg | MS<br>%           | Spike<br>ug/kg | MSD<br>ug/kg | MSD<br>% | RPD              | Limits<br>Rec/RPD |
|-------------------------------|---------------------|---|----------------|-------------|-------------------|----------------|--------------|----------|------------------|-------------------|
| C11-C22 Aromatics<br>(Unadj.) | 112000              |   | 88900          | 265000      | 172* <sup>a</sup> | 91400          | 216000       | 114      | 20               | 40-140/25         |
| C9-C18 Aliphatics             | 129000              |   | 33300          | 254000      | 375* <sup>b</sup> | 34300          | 159000       | 88       | 46* <sup>c</sup> | 40-140/25         |
| C19-C36 Aliphatics            | 35800               |   | 44500          | 106000      | 158* <sup>a</sup> | 45700          | 77000        | 90       | 32* <sup>c</sup> | 40-140/25         |

(a) Outside control limits due to possible matrix interference.

(b) Outside control limits due to high level in sample relative to spike amount.

(c) High RPD due to possible matrix interference and/or sample non-homogeneity.

**Note:** No action taken. MS/MSD % recoveries results and RPD apply to unspiked sample. Unspiked sample from another project.

## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met and/or see below           

No action is taken on MS/MSD results alone to qualify the entire case. However, used informed professional judgment, the data reviewer may use the MS/MSD results in conjunction with other QC criteria and determine the need for some qualification of the data. In those instances where it can be determined that the results of the MS/MSD affect only the sample spiked, the qualification should be limited to this sample alone. However, it may be determined through the MS/MSD results that the laboratory is having a systematic problem in the analysis of one or more analytes, which affects the associated samples.

### 2. MS/MSD – Unspiked Compounds

List the concentrations of the unspiked compounds and determine the % RSDs of these compounds in the unspiked sample, matrix spike, and matrix spike duplicate.

| COMPOUND | CONCENTRATION |    | MSD | %RPD | ACTION |
|----------|---------------|----|-----|------|--------|
|          | SAMPLE        | MS |     |      |        |
|          |               |    |     |      |        |
|          |               |    |     |      |        |
|          |               |    |     |      |        |
|          |               |    |     |      |        |
|          |               |    |     |      |        |
|          |               |    |     |      |        |
|          |               |    |     |      |        |

Criteria: None specified, use  $\%RSD \leq 50$  as professional judgment.

#### Actions:

If the  $\% RSD > 50$ , qualify the results in the spiked sample as estimate (J).

If the  $\% RSD$  is not calculable (NC) due to nondetect value in the sample, MS, and/or MSD, use professional judgment to qualify sample data.

A separate worksheet should be used for each MS/MSD pair.



## DATA REVIEW WORKSHEETS

All criteria were met   X    
Criteria were not met and/or see below           

### VIII. LABORATORY CONTROL SAMPLE (LCS/LCSD) ANALYSIS

This data is generated to determine accuracy of the analytical method for various matrices.

#### 1. LCS Recoveries Criteria

List the %R of compounds which do not meet the criteria

| LCS ID   | COMPOUND | % R | QC LIMIT | ACTION |
|--|----------|-----|----------|--------|
| <u>  LCS_RECOVERY_WITHIN_LABORATORY_CONTROL_LIMITS  </u> |          |     |          |        |
|  |          |     |          |        |
|  |          |     |          |        |
|  |          |     |          |        |
|  |          |     |          |        |

#### Criteria:

- \* Refer to QAPP for specific criteria.
- \* The spike recovery must be between 40% and 140%. Lower recoveries of n-nonane are permissible. If the recovery of n-nonane is <30%, note the nonconformance in the executive narrative. RPD between LCS/LCSD must be < 25%.

#### Actions:

Actions on LCS recovery should be based on both the number of compounds that are outside the %R and RPD criteria and the magnitude of the exceedance of the criteria.

If the %R of the analyte is > UL, qualify all positive results (j) for the affected analyte in the associated samples and accept nondetects.

If the %R of the analyte is < LL, qualify all positive results (j) and reject (R) nondetects for the affected analyte in the associated samples.

If more than half the compounds in the LCS are not within the required recovery criteria, qualify all positive results as (J) and reject nondetects (R) for all target analyte(s) in the associated samples.

#### 2. Frequency Criteria:

Where LCS analyzed at the required frequency and for each matrix (1 per 20 samples per matrix)? Yes or No.

If no, the data may be affected. Use professional judgment to determine the severity of the effect and qualify data accordingly. Discuss any actions below and list the samples affected. Discuss the actions below:

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## DATA REVIEW WORKSHEETS

All criteria were met   X    
Criteria were not met and/or see below           

### IX. FIELD/LABORATORY DUPLICATE PRECISION

Sample IDs:                      -                     

Matrix:        -       

Field/laboratory duplicate samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than laboratory duplicates which measures only laboratory performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field duplicate samples.

| COMPOUND   | SQL | SAMPLE CONC. | DUPLICATE CONC. | RPD | ACTION |
|--|-----|--------------|-----------------|-----|--------|
|  |     |              |                 |     |        |
|  |     |              |                 |     |        |
| No field/laboratory duplicate analyzed with this data package. MS/MSD recoveries results RPD used to assess precision. RPD within laboratory and generally acceptable control limits |     |              |                 |     |        |
|  |     |              |                 |     |        |
|  |     |              |                 |     |        |

#### Criteria:

The project QAPP should be reviewed for project-specific information.  
RPD  $\pm$  30% for aqueous samples, RPD  $\pm$  50 % for solid samples if results are  $\geq$  SQL.  
If both samples and duplicate are  $<5$  SQL, the RPD criteria is doubled.

SQL = soil quantitation limit

#### Actions:

If both the sample and the duplicate results are nondetects (ND), the RPD is not calculable (NC). No action is needed.

Qualify as estimated positive results (J) and nondetects (UJ) for the compound that exceeded the above criteria.

If one sample result is not detected and the other is  $\geq 5x$  the SQL qualify (J/UJ).

**Note:** If SQLs for the sample and duplicate are significantly different, use professional judgment to determine if qualification is appropriate.

If one sample value is not detected and the other is  $< 5x$  the SQL, use professional judgment to determine if qualification is appropriate.

## DATA REVIEW WORKSHEETS

All criteria were met   X    
Criteria were not met and/or see below           

### XI. COMPOUND IDENTIFICATION

The compound identification evaluation is to verify that the laboratory correctly identified target analytes as well as tentatively identified compounds (TICs).

1. Verify that the target analytes were within the retention time windows.

- Retention time windows must be re-established for each Target EPH Analyte each time a new GC column is installed, and must be verified and/or adjusted on a daily basis.
- The n-nonane (n-C9) peak must be adequately resolved from the solvent front of the chromatographic run.
- All surrogates must be adequately resolved from the Aliphatic Hydrocarbon and Aromatic Hydrocarbon standards.
- For the purposes of this method, adequate resolution is assumed to be achieved if the height of the valley between two peaks is less than 25% of the average height of the two peaks.
- The n-pentane (C5) and MtBE peaks must be adequately resolved from any solvent front that may be present on the FID and PID chromatograms, respectively.

1a. Aliphatic hydrocarbons range:

- Determine the total area count for all peaks eluting 0.1 minutes before the retention time (Rt) for n-C9 and 0.01 minutes before the Rt for n-C19.
- Determine the total area count for all peaks eluting 0.01 minutes before the Rt for n-C19 and 0.1 minutes after the Rt for n-C36.

Are the aliphatic hydrocarbons range properly determined?

Yes? or No?

Comments:

1b. Aromatic hydrocarbons range:

- Determine the total area count for all peaks eluting 0.1 minutes before the retention time (Rt) for naphthalene and 0.1 minutes after the Rt for benzo(g,h,i)perylene.
- Determine the peak area count for the sample surrogate (OTP) and fractionation surrogate(s). Subtract these values from the collective area count value.

Are the aliphatic hydrocarbons range properly determined?

Yes? or No?

Comments:

## DATA REVIEW WORKSHEETS

All criteria were met   X    
Criteria were not met and/or see below           

2. If target analytes and/or TICs were not correctly identified, request that the laboratory resubmit the corrected data.
3. Breakthrough determination - Each sample (field and QC sample) must be evaluated for potential breakthrough on a sample specific basis by evaluating the % recovery of the fractionation surrogate (2-bromonaphthalene) and on a batch basis by quantifying naphthalene and 2-methylnaphthalene in both the aliphatic and aromatic fractions of the LCS and LCSD. If either the concentration of naphthalene or 2-methylnaphthalene in the aliphatic fraction exceeds 5% of the total concentration for naphthalene or 2-methylnaphthalene in the LCS or LCSD, fractionation must be repeated on all archived batch extracts.

**NOTE:** The total concentration of naphthalene or 2-methylnaphthalene in the LCS/LCSD pair includes the summation of the concentration detected in the aliphatic fraction and the concentration detected in the aromatic fraction.

Comments: Concentration in the aliphatic fraction < 5% of the total concentration for naphthalene and 2-methylnaphthalene

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4. **Fractionation Check Standard** – A fractionation check solution is prepared containing 14 alkanes and 17 PAHs at a nominal concentration of 200 ng/μl of each constituent. The Fractionation Check Solution must be used to evaluate the fractionation efficiency of each new lot of silica gel/cartridges, and establish the optimum hexane volume required to efficiently elute aliphatic hydrocarbons while not allowing significant aromatic hydrocarbon breakthrough. For each analyte contained in the fractionation check solution, excluding n-nonane, the Percent Recovery must be between 40 and 140%. A 30% Recovery is acceptable for n-nonane.

Is a fractionation check standard analyzed?

Yes? or No?

Comments: Not applicable.

## DATA REVIEW WORKSHEETS

All criteria were met   X    
Criteria were not met and/or see below           

### XII. QUANTITATION LIMITS AND SAMPLE RESULTS

The sample quantitation evaluation is to verify laboratory quantitation results.

In order to demonstrate the absence of aliphatic mass discrimination, the response ratio of C28 to C20 must be at least 0.85. If <0.85, this nonconformance must be noted in the laboratory case narrative.

The chromatograms of Continuing Calibration Standards for aromatics must be reviewed to ensure that there are no obvious signs of mass discrimination.

Is aliphatic mass discrimination observed in the sample? Yes? or No?

Is aromatic mass discrimination observed in the sample? Yes? or No?

1. In the space below, please show a minimum of one sample calculation:

MC45838-2MS      o-terphenyl      RF = 102,400

[ ] = (1535014)/(102,400)

[ ] = 14.99 ppm    Ok

Blank Spike      2-fluorobiphenyl      RF = 92,230

[ ] = (1336918)/(92,230)

[ ] = 14.50 ppm    Ok

## DATA REVIEW WORKSHEETS

2. If requested, verify that the results were above the laboratory method detection limit (MDLs).
3. If dilutions performed, were the SQLs elevated accordingly by the laboratory? List the affected samples and dilution factor in the table below.

| SAMPLE ID | DILUTION FACTOR | REASON FOR DILUTION |
|-----------|-----------------|---------------------|
|           |                 |                     |
|           |                 |                     |
|           |                 |                     |
|           |                 |                     |
|           |                 |                     |
|           |                 |                     |
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|           |                 |                     |
|           |                 |                     |
|           |                 |                     |
|           |                 |                     |
|           |                 |                     |

If dilution was not performed, estimate results (J) for the affected compounds. List the affected samples/compounds:

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